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DOE/NASA/0067-79-6
NASA CR-165313

(NASA-CR-165318) TECHNOLOGY DEVELOPMENT FOR
PHOSPHORIC ACID FUEL CELL POWERPLANT (PHASE
2) Quarterly Report (Energy Research Corp.,
Danbury, Conn.) 49 p HC A03/MF A01 CSCI 10A

N81-22475

Unclass

G3/44 42135

TECHNOLOGY DEVELOPMENT FOR PHOSPHORIC ACID
FUEL CELL POWERPLANT (PHASE II): 7TH QUARTERLY REPORT

LARRY CHRISTNER
ENERGY RESEARCH CORPORATION

JUNE 1980



PREPARED FOR
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
UNDER CONTRACT DEN3-67

FOR
U.S. DEPARTMENT OF ENERGY
ENERGY TECHNOLOGY
DIVISION OF FOSSIL UTILIZATION

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U.S. DEPARTMENT OF ENERGY
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DIVISION OF FOSSIL FUEL UTILIZATION
WASHINGTON, D.C. 20545
UNDER INTERAGENCY AGREEMENT DE-AI-03-79 ET11272

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ENERGY RESEARCH CORPORATION**EXECUTIVE SUMMARY****COMPONENT DEVELOPMENT**

- The use of 1000 grit silicon carbide as received, without cleaning, produces a more porous, faster wicking matrix and has been adopted as the standard material.
- A number of 25 cm² cells with AICMs are performing well in tests and a limiting value has been determined for the amount of electrolyte that can be stored.
- A useful correlation has been found between the degree of a backing's wetproofing and its electrical resistivity.
- Bipolar plates for 350 cm² and 1200 cm² stacks were successfully heat-treated, resulting in decreased hydrogen permeability, reduced electrical resistivity and improved corrosion resistance.

MATERIAL EVALUATION

- The first Tafel slope in composite materials in the potential range of 0.5 to 0.80 V(RHE) most likely corresponds to the electrochemical corrosion reaction of resin.
- The dissolved gases (O₂, N₂, CO₂, and air) have exhibited no effect on the electrochemical corrosion behavior of the Varcum composite.

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- Heat-treatment, i.e., carbonization of the Varcum composite, decreases the corrosion current while increasing the closed porosity.

- The obtained experimental weight loss data of Varcum composite can be represented by the following empirical equation:

$$W_{fs} - W_0 = a (1 - e^{-bt}) - c t$$

This equation also points out the occurrence of a significantly higher corrosion rate in the initial hours of self corrosion.

- The resistance of heat-treated bipolar plates is negligible compared to the current production plates; therefore the employment of heat-treated plates in a 5-cell stack reduces the total resistance of the stack by 58%.

- The contact resistance between the bipolar plate and the backing paper is not significantly affected with increasing temperature in the pressure range of 0.69×10^3 to 1.38×10^3 kPa.

ENDURANCE TESTING

- In 6400 hours of operation, SiC and Mat-1 matrix stacks have operated with comparable performance.

ENERGY RESEARCH CORPORATIONSHORT STACK TESTING

- A performance level of 665 mV/cell at 100 mA/cm² was achieved in a 5 cell, 1200 cm² stack; this is comparable to a 3-cell, 350 cm² stack with similar components.

- The three 5 cell, 1200 cm² stacks started to examine long-term endurance (5000 hours) have completed ~400 hours of testing.

- A 5 cell, 1200 cm² stack operated for almost 3000 hours at an average cell performance (life) of ~0.55 V/cell at 100 mA/cm². Performance of the stack was constant at 0.56 V/cell for almost 2800 hours.

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TASK I. COMPONENT DEVELOPMENT

1.1 MATRIX DEVELOPMENT

Uncleaned SiC appears to be not only acceptable, but beneficial in matrices. As reported previously,* SiC has routinely been cleaned to remove a very small, non-green fraction of the material. The cleaning step is time consuming and whatever small amount of impurities may be present in the non-green fraction, the difference in chemical makeup between clean and unclean SiC is not detectable by emission spectrophotometer. In addition to eliminating a step, the use of unclean SiC produces matrices that are mechanically strong, have high bubble pressures, and are more porous and faster wicking than matrices made with clean SiC. The matrices are strong enough to be cut to shape with a steel rule die, a time-saving step introduced previously. Bubble pressures of these matrices have been measured as high as 210 kPa. The average porosity is 62% vs 51% for standard SiC matrices.

In one test, a matrix made with unclean SiC has wicked significantly faster than one made with clean SiC (Figure I.1). The matrix has performed very well in a number of 25 cm² test cells. Cell 104, built specifically to test the matrix, reached a peak performance of 678 mV at 200 mA/cm² IR free and was still performing well at 1800 hours when terminated (Table I.1). Cells 105, 106, 110, 111, 112 and 113 also contain matrices made with unclean SiC, although their primary purpose was to test AICMs (Table I.1). All cells except No. 112 have performed well and will be discussed further in Section 1.3.

On the basis of all the tests conducted, unclean SiC has been adopted as the standard material for SiC matrices.

* ERC Technical Progress Report DEN3-67, No. 6, Jan.-Mar. 1980.

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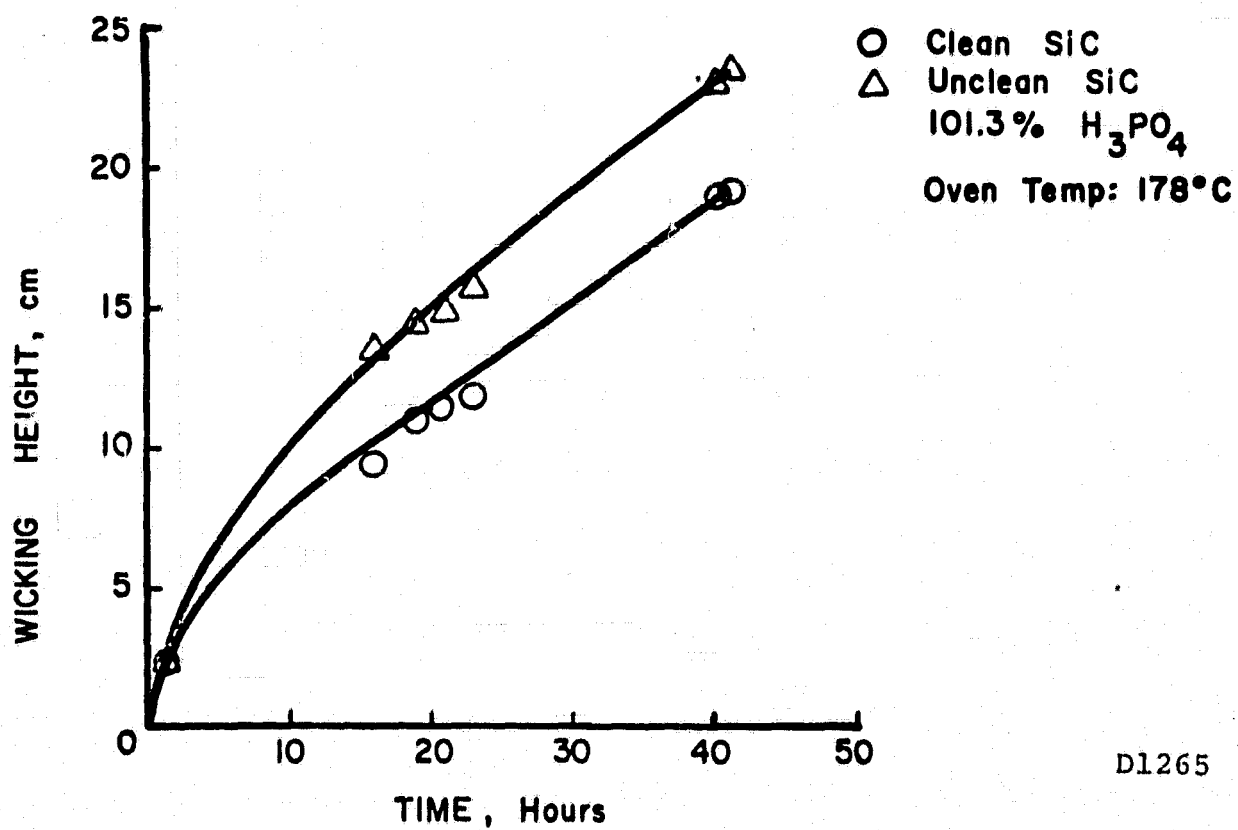


FIGURE I.1 WICKING OF CLEAN vs UNCLEAN SiC MATRICES

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TABLE I.1

CELL TESTING SUMMARY

(Page.1 of 3)

| CELL NO. | 100 | 103 | 105 | 106 |
|--|-----------------|-----------------|-----------------|-----------------|
| TEST OBJECTIVE | AICM | AICM | AICM | AICM |
| <u>CELL CHARACTERISTICS</u> | | | | |
| ANODE | | | | |
| Type | Rolled | Rolled | Rolled | Rolled |
| TFE, % | 40 | 40 | 40 | 40 |
| Loading, mg Pt/cm ² | 0.35 | 0.30 | 0.33 | 0.33 |
| CATHODE | | | | |
| Type | Rolled | Rolled | Rolled | Rolled |
| TFE, % | 40 | 40 | 40 | 40 |
| Loading, mg Pt/cm ² | 0.56 | 0.65 | 0.62 | 0.62 |
| MATRIX | Sic | Sic | Sic | Sic |
| TFE, % | 4 | 4 | 4 | 4 |
| Porosity, % | - | - | 62 | 62 |
| Thickness, cm | 0.015 | 0.020 | 0.023 | 0.023 |
| Sintering | 15 min. @ 330°C | 15 min. @ 330°C | 15 min. @ 330°C | 15 min. @ 330°C |
| ANODE BACKING % FEP | 27 | 25 | 35 | 35 |
| CATHODE BACKING % FEP | 36 | 37 | 39 | 39 |
| <u>PEAK PERFORMANCE, mV</u> IR-Free | | | | |
| AIR - 100 mA/cm ² | 671 | 665 | 688 | 681 |
| 200 mA/cm ² | 648 | 605 | 643 | 632 |
| O ₂ - 100 mA/cm ² | 733 | 726 | 751 | 746 |
| 200 mA/cm ² | 690 | 675 | 711 | 704 |
| O ₂ GAIN - 100 mA/cm ² | 62 | 60 | 57 | 64 |
| 200 mA/cm ² | 42 | 70 | 70 | 70 |
| <u>PRESENT PERFORMANCE</u> | | | | |
| AIR - 200 mA/cm ² | * | * | 631 | 620 |
| <u>CELL LIFE, hours</u> | 1464 | 2856 | 2304 | 2256 |

* Test Terminated

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TABLE I.1

CELL TESTING SUMMARY

(Page 2 of 3)

| CELL NO. | 110 | 111 | 112 | 113 |
|--|-----------------|-----------------|-----------------|-----------------|
| TEST OBJECTIVE | AICM | AICM | AICM | AICM |
| <u>CELL CHARACTERISTICS</u> | | | | |
| ANODE | | | | |
| Type | Rolled | Rolled | Sheet Mold | Rolled |
| TFE, % | 35 | 35 | 40 | 35 |
| Loading, mg Pt/cm ² | 0.25 | 0.15 | 0.35 | 0.15 |
| CATHODE | | | | |
| Type | Rolled | Rolled | Rolled | Rolled |
| TFE, % | 40 | 40 | 40 | 40 |
| Loading, mg Pt/cm ² | 0.62 | 0.62 | 0.56 | 0.66 |
| MATRIX | Sic | Sic | Sic | Sic |
| TFE, % | 4 | 4 | 4 | 4 |
| Porosity, % | 62 | 62 | 64 | 59 |
| Thickness, cm | 0.023 | 0.023 | 0.020 | 0.023 |
| Sintering | 15 min. @ 330°C | 15 min. @ 330°C | 15 min. @ 330°C | 15 min. @ 330°C |
| ANODE BACKING % FEP | 36 | 39% PTFE | 15% PTFE | 39% PTFE |
| CATHODE BACKING % FEP | 39 | 39 | 38 | 36 |
| <u>PEAK PERFORMANCE, mV</u> IR-Free | | | | |
| AIR - 100 mA/cm ² | 688 | 705 | 655 | 702 |
| 200 mA/cm ² | 632 | 650 | 579 | 634 |
| O ₂ - 100 mA/cm ² | 754 | 765 | 715 | 772 |
| 200 mA/cm ² | 713 | 720 | 659 | 724 |
| O ₂ GAIN - 100 mA/cm ² | 62 | 60 | 60 | 60 |
| 200 mA/cm ² | 68 | 70 | 80 | 90 |
| <u>PRESENT PERFORMANCE</u> | | | | |
| AIR - 200 mA/cm ² | 621 | 650 | 579 | 634 |
| <u>CELL LIFE, hours</u> | 768 | 480 | 264 | 120 |

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TABLE I.1

CELL TESTING SUMMARY

(Page 3 of 3)

| CELL NO. | 1345 | 104 | 114 |
|--|--|-----------------------|-----------------|
| TEST OBJECTIVE | Large Sheet Mold Electrodes & AICM Control | Unclean Sic Matrix | AICM |
| <u>CELL CHARACTERISTICS</u> | | | |
| ANODE | | | |
| Type | Sheet Mold | Sheet Mold | Rolled |
| TFE, % | 40 | 40 | 35 |
| Loading, mg Pt/cm ² | 0.45 | 0.32 | 0.15 |
| CATHODE | | | |
| Type | Sheet Mold | Rolled | Rolled |
| TFE, % | 40 | 40 | 40 |
| Loading, mg Pt/cm ² | 0.50 | 0.62 | 0.62 |
| MATRIX | | | |
| | Kynol | Sic | Sic |
| TFE, % | - | 4 | 4 |
| Porosity, % | - | 62 | 63 |
| Thickness, cm | 0.048 | 0.023 | 0.023 |
| Sintering | - | 15 min. @ 330°C | 15 min. @ 330°C |
| ANODE BACKING % FEP | 35 | 34 | 39% PTFE |
| CATHODE BACKING % FEP | 34 | 39 | 37 |
| <u>PEAK PERFORMANCE, mV</u> IR-Free | | | |
| AIR - 100 mA/cm ² | 733 | 710 | 711 |
| 200 mA/cm ² | 710 | 678 | 662 |
| O ₂ - 100 mA/cm ² | 793 | 780 | 771 |
| 200 mA/cm ² | 775 | 731 | 732 |
| O ₂ GAIN - 100 mA/cm ² | 60 | 50 | 60 |
| 200 mA/cm ² | 60 | 60 | 70 |
| <u>PRESENT PERFORMANCE</u> | | | |
| AIR - 200 mA/cm ² | * | * | 662 |
| <u>CELL LIFE, hours</u> | 8400 | 1800 | 96 |

* Test Terminated

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The use of 1 μm particle size SiC has been re-evaluated. It was hoped that reducing the particle size from the standard 7 μm mean particle diameter would result in matrices of higher bubble pressure. The smaller particle size SiC, when last evaluated two years ago, produced cracked, crumbly and totally unacceptable matrices. Since the smaller particle size has many more particles in a given volume of matrix, it takes proportionately more binder to hold them together. To eliminate cracking, it was necessary to add so much PTFE that the matrix lost its wicking ability. It had been hoped that improvements in matrix formulation, casting and sintering techniques over the last two years would permit production of good quality matrices with 1 μm SiC and acceptable PTFE levels. Such was not the case. Matrices made with the standard 4% PTFE cracked and crumbled to such an extent that further work with 1 μm SiC is not considered worthwhile.

1.2 COMPONENT SCALE-UP

Production of rolled and sheet mold electrodes, and SiC matrices for use in 1200 cm^2 stacks is continuing without difficulty. The three 350 cm^2 and nine 1200 cm^2 stacks built during this quarter are reported in detail in Task IV.

1.3 DEFINITION AND CONTROL OF ELECTROLYTE VOLUME CHANGES

Development of selectively wetproofed backings for use as acid inventory control members (AICMs) is continuing. A large number of AICMs have been produced and tested for acid storage capacity, acid transfer to the catalyst layer and for performance in test cells. The uniformity of AICMs appears to be improving, because the range of values obtained for acid pick-up generally falls within 10% for a given AICM.

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A PTFE emulsion has been evaluated for use in selectively wetproofed backings and appears to work as well as the FEP emulsion normally used. One AICM wetproofed with PTFE had an average acid pick-up of 13.5×10^{-3} cc/cm². This is equivalent to 5.2 cc capacity per anode in a 350 cm² stack and 15.1 cc in a 1200 cm² stack. This same AICM has been used as the anode in Cells 111, 113 and 114, which are all doing well despite a somewhat low catalyst loading (Table I.1). Their average performance is almost 650 mV at 200 mA/cm², IR free. Approximately 35% of the surface area of the backing (facing the catalyst) was wetproofed. Since these cells appear to be working well (at least to the extent that their performance does not seem to have been hurt by the AICMs), that level of wetproofing would appear to be acceptable. Previous AICM test cells have also been in the range of 30% to 45% wetproofed. It is possible that with improved wetproofing patterns and techniques, the amount of wetproofing necessary could be reduced further. However it is obvious that in the extreme case, the non-wetproofed area could not be increased by more than 50% of its present area. This means that, barring a significant increase in backing thickness, no more than 20×10^{-3} cc/cm² of acid could be stored in an AICM produced by the present technique. Scaled up to stack size, the limiting acid storage capacity per cell (in the AICM) would be approximately 7.7 cc in a 350 cm² stack and 22.4 cc in a 1200 cm² stack.

Two of the 25 cm² AICM test cells that were running at the time of the last quarterly report have been terminated. Cells 100 and 103 (Table I.1) both exhibited deterioration in performance, probably due to flooding of the anode (AICM) backing. Although O₂ gains remained low, voltage readings dropped rapidly as the load was increased. If the catalyst layer itself were being flooded, O₂ gains would be expected to increase due to diffusion polarization. The kind of deterioration found, therefore, points to a loss of active

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catalyst area caused by backing flooding. When the cells were run in reverse, the drop in performance was too great to be attributed to the difference in catalyst loading between the anode and cathode; thus the anode is clearly the problem electrode. Cells 105 and 106 are showing similar signs of deterioration after running for more than 2200 hours (Table I.1).

Five new AICM test cells have been built and, with one exception, are performing steadily at acceptable levels (Table I.1). Cell 110 shows a slight drop in performance, but it is too little and too early to pinpoint a definite cause. Cells 111, 112, 113 and 114 are all running at peak performance and all have AICMs prepared with PTFE instead of FEP. Cell 112 has a low PTFE content AICM (15 wt%) and a sheet mold anode (Table I.1). This is the first time at ERC that a sheet mold catalyst layer has been combined with an AICM backing. The cell is producing only 579 mV at 200 mA/cm², IR free. The low performance appears to be due to the low degree of AICM wetproofing, possibly aggravated by catalyst material being sucked further into the backing than would occur with a completely wet-proofed backing.

Cell 1345, as previously reported, was used as a control in an experiment designed to determine the effect of acid deprivation on cells with AICMs. The cell also had a very high and unstable internal resistance which, after being corrected, indicated a very high level of performance. After more than 7500 hours of running under adverse conditions, the cell peaked at 710 mV at 200 mA/cm², IR free. This performance must be considered an anomaly and several attempts were made to determine the cause of the extremely high IR (56 mΩ at one point). When the cell was rebuilt with completely new hardware (plates, current collectors, etc.), the cell's IR dropped to 16 mΩ and the IR-free performance was 605 mV at

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200 mA/cm². The cell could not be studied in great detail because its matrix (Kynol) was damaged during disassembly causing the cell performance to begin deteriorating rapidly. Therefore it must be assumed that the cause of the cell's high IR was hardware-related, although there were no obvious signs of corrosion or other degradation.

1.4 BACKING PAPER TECHNOLOGY

As previously reported, there is considerable variability in the wetproofing of the large carbon paper backings, probably as a result of the method of FEP treatment. Techniques to produce more uniform wetproofing of backings are being evaluated. However in the meantime, an attempt has been made to relate the degree of wetproofing (indicative of the amount of FEP contained) to the electrical resistivity of the backing. As expected, the resistivity of the backing increases in direct proportion to the degree of wetproofing since FEP is an excellent insulator.

A 2270 cm² backing originally intended for a sheet mold electrode was used in the test. The prewashed and single dipped backing contained 25.5% FEP overall. The backing had been tipped from side to side during draining which might have been expected to produce high FEP concentrations at the two corners where draining occurred, low concentrations at the corners which drained into the other two, and an intermediate concentration in the center. The results from the resistivity and acid pickup tests indicate this to be the case. Table I.2 lists the apparent resistivity of 25 cm² pieces cut from the large backing. The term "apparent resistivity" is used because there appears to be a significant contact resistance factor in the measurement of the resistance. This contact resistance is discussed further in Section 2.2.1. Although the actual resistivities may vary from the values calculated, the relationship between samples is thought to be valid. Silver electrodes were used to take the measurements and an air press

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was used to ensure good contact between the silver and the carbon paper. The resistance was measured at a series of different pressures, but only those values obtained at the greatest pressure (433 kPa) are in the table. Again, although all of the numbers were higher at lower pressures, the relationship between the samples did not vary. Table I.2 also lists the weight of acid the samples picked up after being floatfilled for 19 hours in 100.7% H_3PO_4 at 178°C. Two sets of numbers are plotted in Figure I.2 and analyzed by linear regression methods. The locations of the samples on the backing are also shown in the figure. Although the data points actually fit well on a curve, the correlation coefficient, a measure of how close the data points are to a straight line drawn through them, is 0.956. This high a correlation (a perfect fit would be 1.000) indicates a very strong relationship between the apparent resistivity of the backing paper and its degree of wetproofing (and, hence, FEP content). Possibly this relationship could be used as a mutual indicator of either degree of wetproofing or backing

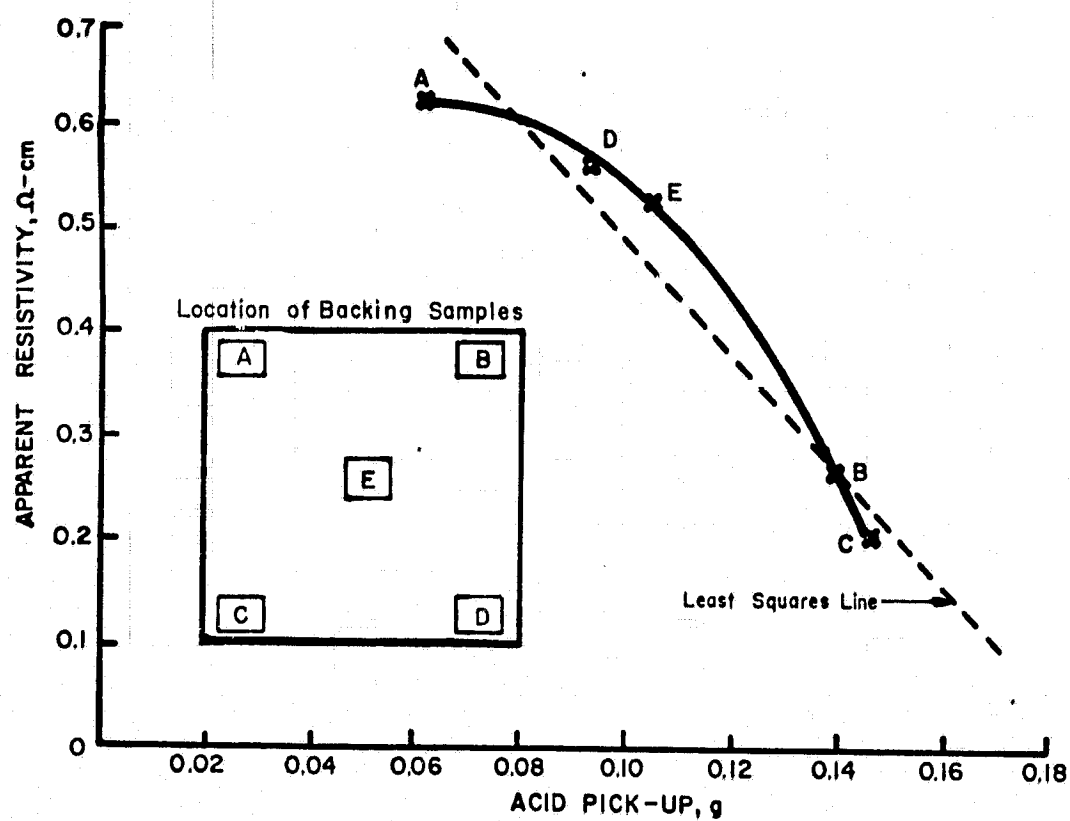
TABLE I.2

APPARENT RESISTIVITY AND ACID PICK-UP OF CARBON BACKING PAPER

| SAMPLE [*] | APPARENT RESISTIVITY, Ω -cm | ACID PICK-UP, g |
|---------------------|---------------------------------------|--------------------|
| A | 0.620 | 0.061 |
| B | 0.267 | 0.139 |
| C | 0.203 | 0.147 |
| D | 0.562 | 0.093 |
| E | 0.525 | 0.105 |

* See location map in Figure I.2.

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FIGURE I.2 BACKING RESISTIVITY vs ACID PICK-UP

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resistivity. The degree to which resistivity varies in a backing could have important implications with respect to the uniformity of current density within a cell, particularly in the case of a selectively wetproofed AICM. This work will be continued.

The carbon backing paper has been found to contain microscopic inorganic particles of zinc and other metals as reported previously. A black, particulate material has also been observed to wash out of the backings during FEP treatment. For this reason a number of backings have been prewashed in a dilute FEP suspension in an attempt to remove this material before the actual wetproofing treatment. The particulate residue from the prewash and the first dip liquids of several of these backings have been analyzed by emission spectroscopy to see if the inorganic materials are, in fact, being washed out of the backing paper. The analyses show that although more total material is being removed in the prewash than in the subsequent dipping procedure, more of the inorganics (i.e., non-carbon particulates) are being removed during the final dipping. This could be caused by the greater amount of wetting agent present in the FEP suspension used for the wetproofing treatment than in the dilute, prewash suspension. In addition to carbon, the prewash residue showed traces of titanium and silicon (0.005 to 0.010%). The residue of the final dipping liquid showed small amounts of titanium and silicon (0.01 to 0.10%) and traces of iron, calcium and magnesium. No zinc was detected in either sample, although X-ray microanalysis showed it to be the most prevalent metal contained in the backing. This could mean that the zinc is in some way bound to the carbon fibers. More quantitative work will be performed to determine whether the metallic contaminants found in the backing paper are present in the finished backing in sufficient quantities to poison the catalyst.

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1.5 BIPOLAR PLATE TECHNOLOGY

1.5.1 Molding

The 1200 and 350 cm² bipolar plates were successfully molded with Reichhold Chemicals' Varcum 29-703 resin and Asbury A-99 graphite. The molding characteristics of the compound were satisfactory compared to current production materials and other experimental compounds. No significant difference was observed between Varcum 24-655 and Varcum 29-703 resins during preforming and molding. But the preforms of both Varcum resins were noticeably different from Colloid resin preforms: Varcum resin preforms were denser but not as strong as the Colloid resin preforms. Additions of Asbury 850 graphite (a flake graphite used with the Colloid molding compound) to Varcum molding compounds did not improve its strength. Rejection rates for forty-six 1200 cm² bipolar plates made with Varcum resin did not differ significantly from current rejection rates, but low density areas in the corners of some plates were observed for a second series of molding trials. The current Varcum molding compounds are acceptable for bipolar plate production, but require more careful preforming and handling procedures than the Colloid 8440 molding compound.

1.5.2 Heat Treatment

A retort has been completed and used to heat treat sixteen 1200 and 350 cm² bipolar plates (Varcum 29-703 and Varcum 24-655 resins). The plate compositions were 32 or 25 wt% resin and Asbury A-99 graphite. The plates shrank uniformly during the heat treatment: $2.5 \pm 0.5\%$ shrinkage for 2.5 wt% resin and $4.0 \pm 0.5\%$ shrinkage for 32 wt% resin.

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The 32 wt% resin plates lost 12 to 14% of their weight. More testing is required to quantify the hydrogen permeability, but initial testing indicated reductions in hydrogen permeability after heat treatment of a 32 wt% resin plate.

Two fuel cell stacks were built with the heat treated 32 wt% Varcum 24-655/68 wt% A-99 bipolar plates: a three cell, 350 cm² stack and a five cell, 1200 cm² stack (refer to Task IV for performance information). Table II.5 contains internal resistance information for a stack with heat treated plates.

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TASK II MATERIAL EVALUATION

2.1 COMPONENT CORROSION RESISTANCE

2.1.1 Electrochemical Corrosion Measurements

A. Polarization study of a Varcum Composite in Different Environments:

The electrochemical corrosion behavior of 32% Varcum 24-655 was studied in 100 to 102% H_3PO_4 at $191^\circ C$ employing different environments. The following gases were bubbled through the electrochemical polarization cell for about 4 hours before starting polarization of the electrode.

- Oxygen (O_2)
- Nitrogen (N_2)
- Carbon Dioxide (CO_2)
- Air

The corresponding polarization curves are shown in Figure II.1. All curves appeared to be similar in nature in the potential range of 0.60 to 1.0V (RHE), while in the lower potential range of 0.45 to 0.575V (RHE) some scattered points were observed. This scattering may be partly due to the oxidation of some impurities at lower potential. The kinetic parameters corresponding to the corrosion reactions in various environments were consistent with those already reported* for the Varcum composite sample. These parameters are tabulated in Table II.1.

* DEN3-67 No. 6, Jan.-Mar. 1980.

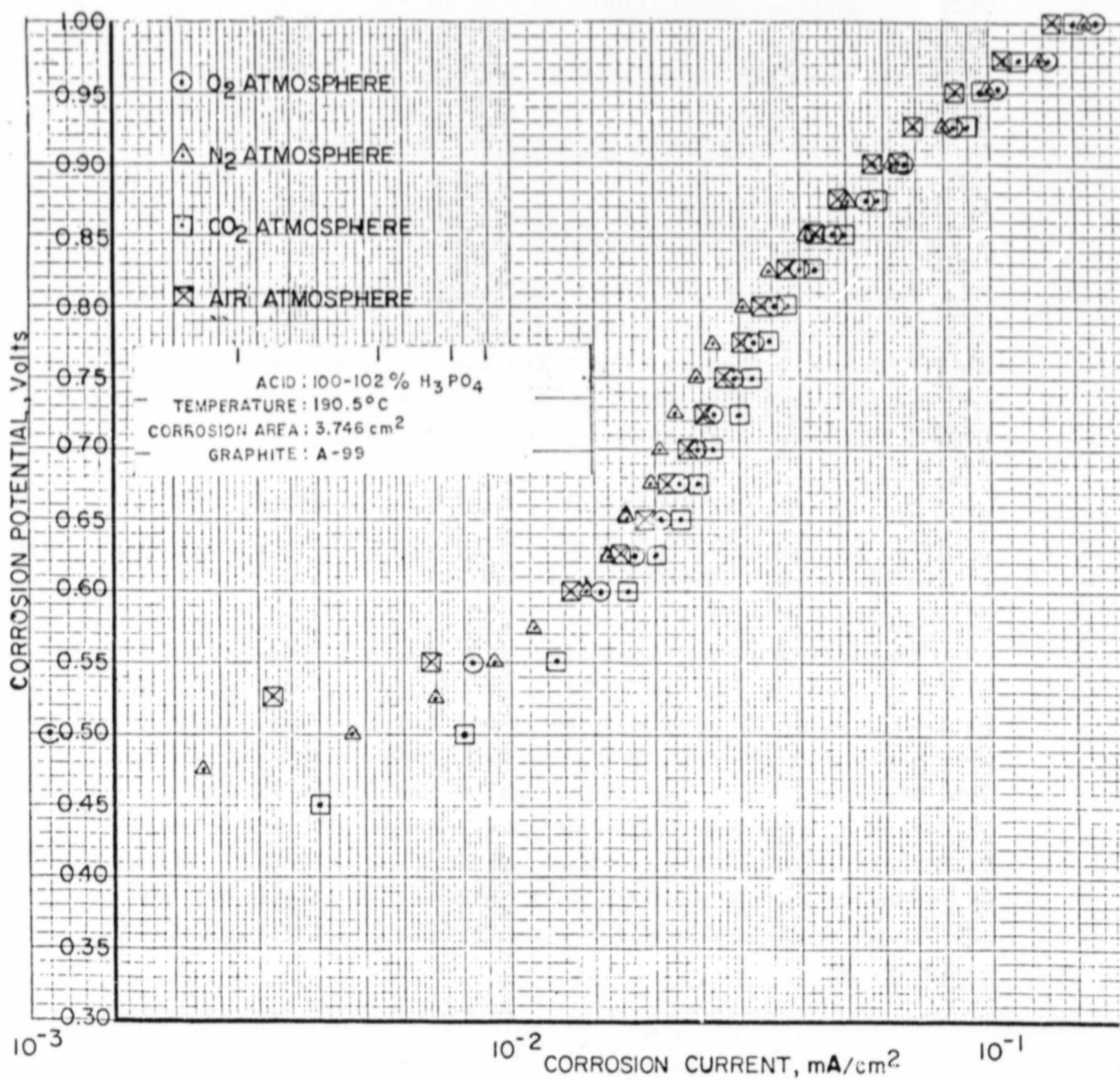


FIGURE II.1 EFFECT OF ENVIRONMENT ON THE ELECTROCHEMICAL CORROSION OF A 32% VARCUM 24-655 SAMPLE

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TABLE II.1

KINETIC PARAMETERS OF VARCUM COMPOSITE CORROSION REACTION IN
DIFFERENT ENVIRONMENTS

| ENVIRONMENTAL GASES | POTENTIAL RANGE BETWEEN 0.8 AND 1.0V (RHE) | | |
|------------------------|--|--|---|
| | EXCHANGE CURRENT DENSITY, i_0 , A/cm ² | TAFEL SLOPE $\frac{RT}{\alpha_2 F} \times 2.303$ mV/decade | TRANSFER COEFF. $\frac{1}{\alpha_2}$ |
| CO ₂ | 1.5×10^{-7} | 299 | 3.2 |
| N ₂ | 0.5×10^{-7} | 253 | 2.7 |
| O ₂ | 0.7×10^{-7} | 267 | 2.8 |
| Air | 1.06×10^{-7} | 290 | 3.1 |

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B. Relationship of Electrochemical and Self Corrosion Studies to Fuel Cell Bipolar Plate Corrosion:

In phosphoric acid fuel cells, the cathode potential stays in the vicinity of 0.6 to 0.95V (RHE) depending on cell operating conditions. Therefore the side of the bipolar plate which is in contact with the cathode gets polarized to this potential when it comes in contact with the bulk electrolyte. Under a similar situation, the bipolar plate side which is in contact with the anode would be polarized to the fuel cell anode potential, 0 to 0.015V (RHE).

The experimentally observed rest potentials of different types of bipolar plate materials, in 100 to 102% H_3PO_4 , and in the temperature range of 135 to 190°C, were between 0.3 and 0.4V (RHE). Using the polarization plot of the 32% Varcum 24-655/68% A-99 graphite composite, the electrochemical corrosion current at the rest potential (0.35V [RHE] at 189°C) was found to be 1.8×10^{-7} A/cm². The corresponding weight loss rate† would be 0.02 µg/cm² hr, which is significantly lower than the previously reported‡ self corrosion rate of this sample (0.125 µg/hr cm²).

† Composite weight loss rate = $\frac{i \times 60 \times 60 \times 10^6 \times 12}{4F}$ µg/cm² hr.

‡ DEN3-67, No. 6, Jan.- Mar. 1980.

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The electrochemical corrosion rates of several composites (including heat treated 25% Varcum 24-655) evaluated at 0.9V (RHE) are compared with their self corrosion rates in Figure II.2'. The electrochemical corrosion rate was found to be more than 100 times higher in most of the composite materials; at the rest potential the electrochemical corrosion rate is negligible. Therefore self corrosion is the dominant corrosion process on the anode side of the bipolar plate; while on the cathode side, electrochemical corrosion which is significantly high at cathode potential is the rate controlling process.

C. Comparison of Electrochemical and Self Corrosion Processes of Bipolar Plates:

It has been previously documented* that the electrochemical corrosion of the cathode plates at fuel cell operating potential is much more severe than the self corrosion (corrosion rate measured in acid immersion tests) of the anode plate. Etching patterns were also observed to be different and the possibility of the existence of different corrosion mechanisms was raised. In this study, surfaces of the fresh (control), acid immersed and electrochemically corroded samples were investigated via ESCA (electron spectroscopy for chemical analysis) for further understanding of the corrosion processes discussed. The surface compositions of the elements detected via ESCA spectra in the 0 to 250 eV region, (estimated and expressed as atom percent) are listed in Table II.2. The ESCA survey spectra of all the samples indicated that P, S and Si were present as phosphates, sulphates, and mostly a Si-O type bonding. For the acid immersed sample, Si was detected as Si-O and SiO₂ bonding and the ratio between silica and silica oxide was found to be 3:1. (Note that the high Si content of the acid immersed sample is related to the corrosion of the Pyrex glass cover used in ERC self corrosion cells.) The amount of O present and not bound to P, S, and Si was used to evaluate the C/O atom ratio, as reported in the table.

* DEN 3-67, No. 6, Jan.- Mar. 1980.

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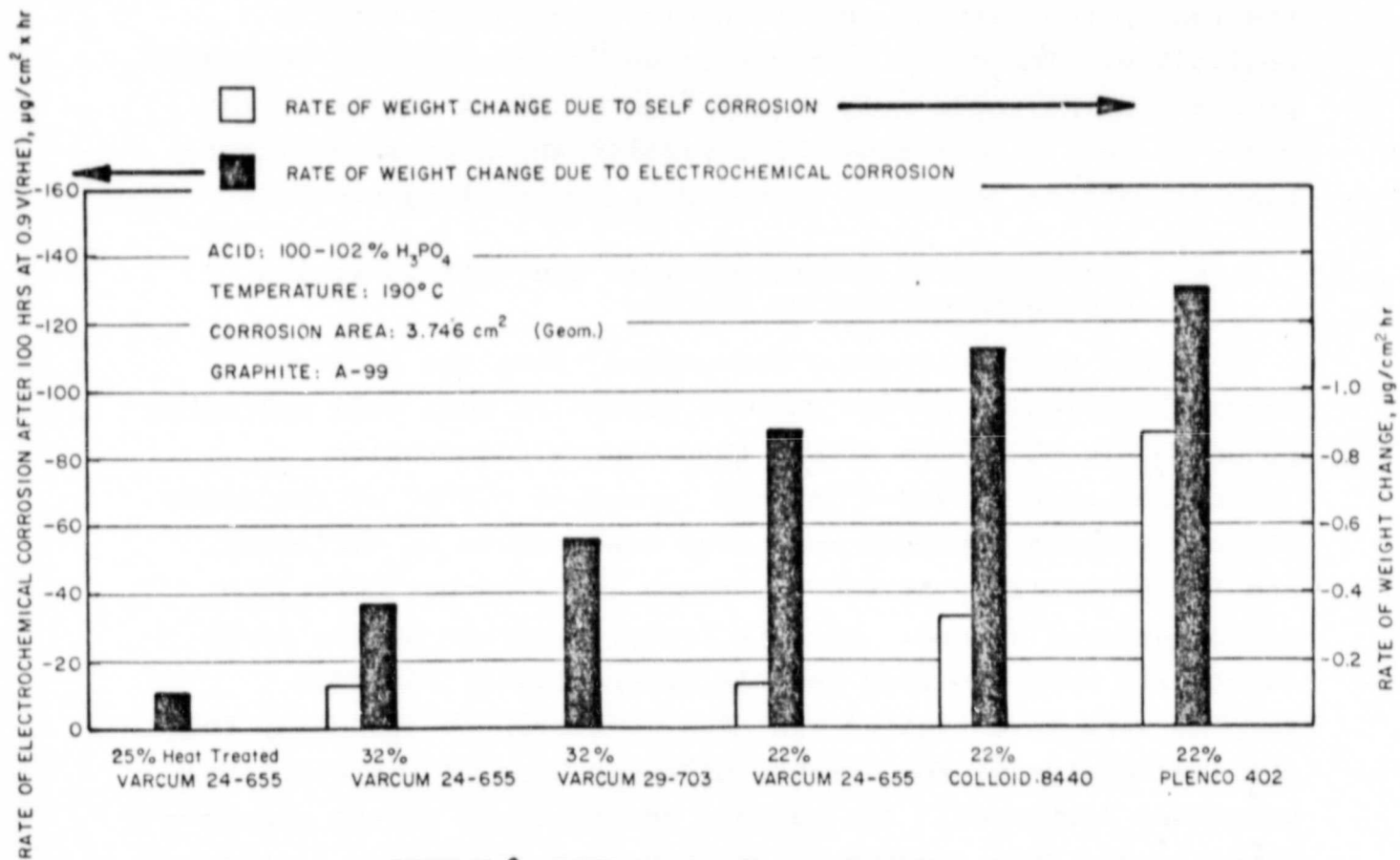


FIGURE II.2 ELECTROCHEMICAL AND SELF-CORROSION
OF DIFFERENT SAMPLES AT 0.9V (RHE) AND 190°C

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TABLE II.2

SURFACE ELEMENTAL COMPOSITIONS OF 32% VARCUM COMPOSITE ESTIMATED FROM ESCA SPECTRA*

| SAMPLE TREATMENT | ELEMENTS | | | | | | | | | C/O ATOM RATIO |
|---------------------------|----------|------|------|-----|-----|-----|-----|-----|-----|-------------------|
| | C | O | Si | N | F | P | S | Cl | Na | |
| Fresh | 80.0 | 13.0 | 2.3 | 3.2 | 0.3 | 0.4 | 0.3 | 0.3 | 0.5 | 10.1 |
| Acid Corroded† | 51.0 | 30.0 | 18.0 | 1.0 | 0.5 | † | † | † | † | 6.8 |
| Electrochemical** Cell | 92.0 | 7.2 | 0.1 | 0.7 | † | 0.3 | † | 0.2 | † | 15.6 |

* Expressed as atom % for the detected elements.

† No observation of a signal.

‡ 32% Varcum 24-655 composite was immersed in 100 to 102% acid at 185°C for 1000 hours and the absorbed acid was removed via Soxhlet extraction.

** 32% Varcum 24-655 composite corroded in an electrochemical cell at 0.9V(RHE) in 100 to 102% H_3PO_4 at 191°C for 430 hours and the acid was removed via Soxhlet extraction.

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The different values of C/O atom ratio, information from SEM photographs, and dissimilarity between the C(1S) spectra for the self-and electrochemically-corroded samples illustrate that the modes of acid attack and electrochemical corrosion are different. For further study of the involved mechanisms, other supplementary analyses (such as AES, IR-spectroscopy, total C, H, and O analysis, etc.) are required for both corroded samples and corrosion products. In support of the previous observation that the acid absorption is totally a physical process* the ESCA analysis of both acid-attacked and electrochemically-corroded samples revealed no change in the structural features of P content of the samples.

D. Effect of Non-uniform Distribution of Graphite within Large Bipolar Plate Materials:

To comprehend the graphite distribution effect on the corrosion rate of a bipolar plate, a cross sectional corrosion study of a 12 in. x 17 in. 0-0 bipolar plate (33% Colloid 8440 composite) was conducted at 190°C. The samples from various regions of an 0-0 plate were cut out and potentiostatically corroded in the electrochemical corrosion cells in 100 to 102% H₃PO₄ at 0.9V (RHE) and 190°C.

The positions of the evaluated samples on the 0-0 bipolar plate and their corrosion rates are documented in Figure II.3. The following observations are significant:

- The sample examined from the center of the plate showed about an order of magnitude higher corrosion rate than samples from the side and corner.
- Different corrosion rates determined for the different regions of the plate indicated non-uniformity and irregularity of the plate.

The higher corrosion rate for the center sample was probably due to lower resin content and higher true acid exposed

* DEN3-67, No. 4, July - Sept. 1979.

Material: O-O Plate
(33 % Colloid 8440 Composite)
100-102 % H_3PO_4
Temperature: 190°C
Controlled Potential: 0.90V(RHE)

Corresponding corrosion rates of samples
are shown in the respective blocks in mA/cm^2

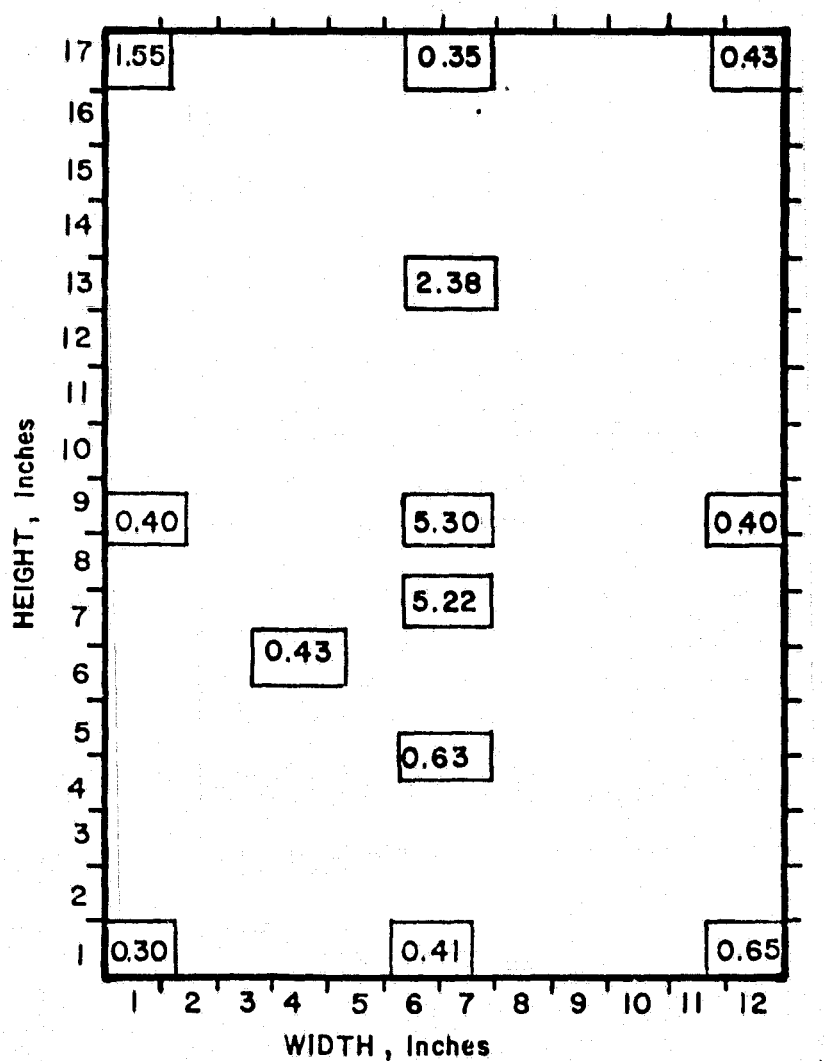


FIGURE II.3 CROSS SECTIONAL CORROSION STUDY
OF O-O PLATE

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area. Attempts are underway to produce bipolar plates with more uniformly distributed graphite and resin contents.

E. Porosity Measurement of Heat Treated and Regular Composite Materials:

Experiments were performed to determine whether the reported* difference in corrosion rates of heat-treated and regular samples is related to different true acid exposed areas.

The void or pore volume which is related to true acid exposed area was estimated by boiling weighed samples (25% Varcum 24-655 composite and heat-treated 25% Varcum 24-655 composite) in water. After the air in the pores was displaced, the samples were superficially dried in air and weighed. The specific pore volumes of these samples determined from the pore volume (i.e., weight gain/density of H₂O) divided by initial weight of the samples are compared in Table II.3. It is obvious from the data that the heat-treated 25% Varcum 24-655 composite had about an order of magnitude higher porosity than the regular 25% Varcum 24-655 composite. It is therefore apparent that heat-treatment of the bipolar plate materials improves the corrosion resistance characteristics of the materials and simultaneously increases the porosity of the materials as well.

2.1.2 Self Corrosion Measurement

Higher Rate of Weight Loss is Present During Initial Hours of Acid Immersion:

Weight loss measurements of several composites (in the time interval of 150 to 2000 hours) were reported to be linear

* DEN3-67, No.6, Jan.- Mar. 1980.

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TABLE II.3

POROSITY COMPARISON OF HEAT-TREATED AND REGULAR
COMPOSITE MATERIALS

| COMPOSITE MATERIAL | DENSITY, g/cc (P) | GEOMETRIC VOLUME OF SAMPLE, cc (V) | INITIAL WT, g (W _O) | WT. GAINED, mg (W _F - W _O) | PORE VOLUME OF THE SAMPLES, cc/g $\frac{(W_F - W_O)/P_{H_2O}}{= V_P W_O}$ | PORE FRACTION $E = \frac{V_P}{V}$ |
|---|-----------------------------|--|---|---|--|--------------------------------------|
| 25% Regular Varcum 24-655 | 1.8205 | 1.06 | 1.9153 | 5.2 | 2.7×10^{-3} | 0.0025 |
| 25% Heat- Treated Varcum 24-655 | 1.9646 | 0.973 | 1.8005 | 51.6 | 2.8×10^{-2} | 0.029 |

A-99 Graphite was used in the composite materials.

W_f = final wt. of the sample.

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with time of acid immersion*. These straight lines did not pass through the origin, raising the possibility that a higher rate of weight loss may be present during the early hours of acid immersion. In this study, the weight loss characteristics of the 25% Varcum 24-655, 75% A-99 graphite composite (presently used in manufacturing fuel cell bipolar plates) was studied in the time interval of 5 to 2500 hours to elucidate the corrosion rate of initial hours.† The weight loss data agree reasonably well with the following empirical equation (Figure II.4):

$$W_{fs} - W_o = a (1 - e^{-bt}) - ct \quad (1)$$

Where,

W_o = initial weight of the sample, mg/cm²

W_{fs} = final weight after self corrosion and Soxhlet extraction, mg/cm²

t = acid immersion time, hours.

The least square estimates of the parameters a , b and c are 0.273 mg/cm², 0.7 hr⁻¹ and 2×10^{-4} mg/cm², respectively. The self corrosion rate of this sample therefore can be expressed as:

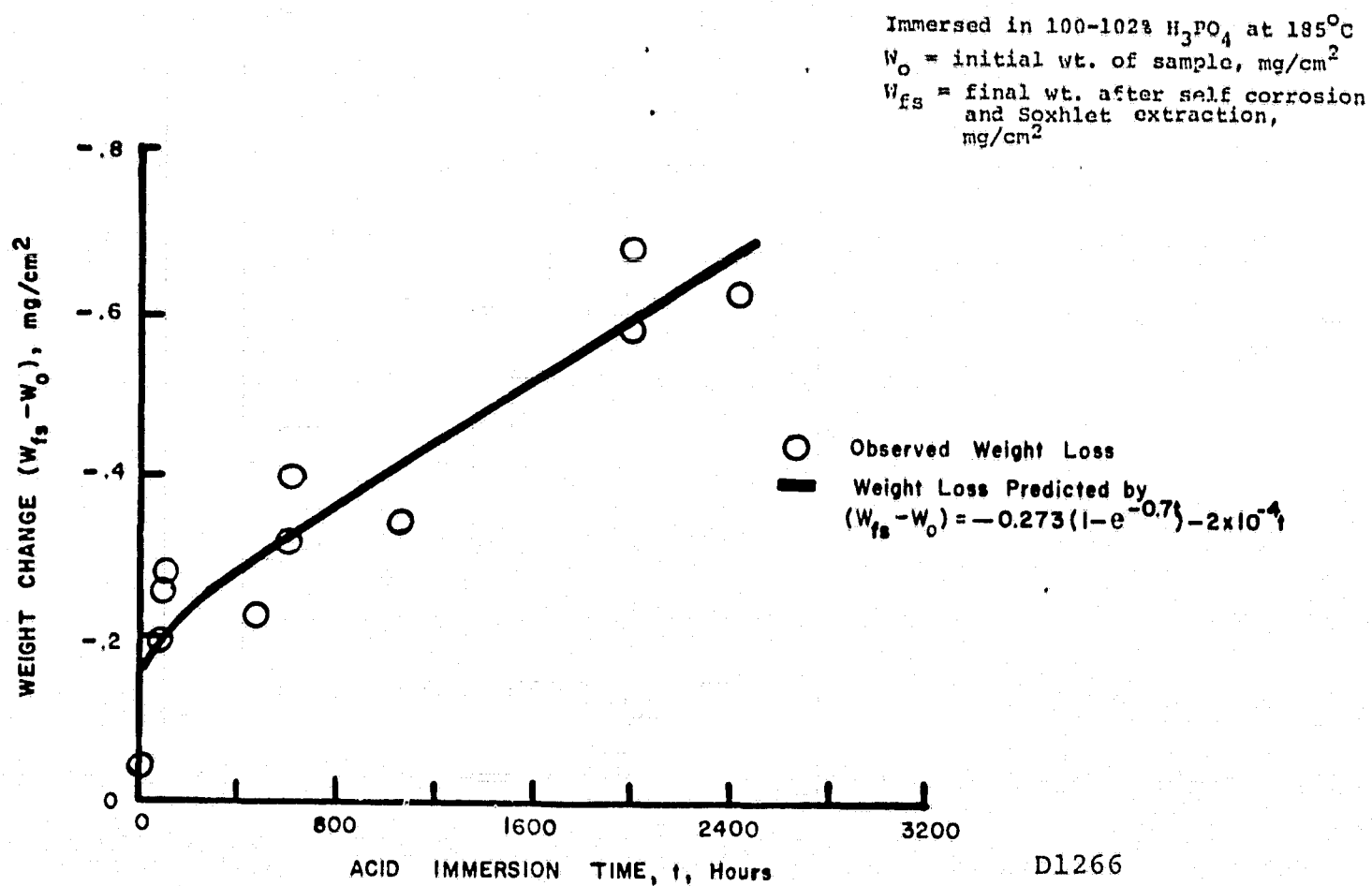
$$\begin{aligned} \frac{d(W_{fs} - W_o)}{dt} &= a \times b e^{-bt} - c \\ &= -0.19 e^{-0.7t} - 2 \times 10^{-4} \end{aligned} \quad (2)$$

Equation 2 reveals that the self corrosion rate after about 15 hours of acid immersion is only 0.2 µg/cm² hr and is also independent of aging time. Note that the self corrosion rate at initial hours is significantly higher and at $t \rightarrow 0$ the corrosion rate is about 0.19×10^3 µg/cm² hr, which is about 3 orders of magnitude larger than the rate observed after 15 hours of acid immersion. Interestingly, the acid absorption

* DEN3-67, No. 6, Jan. - Mar. 1980.

† The scatter of experimental points is probably related to the non-uniformity of the samples used in this study.

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FIGURE II.4 COMPARISON OF OBSERVED AND PREDICTED WEIGHT LOSS
 IN 25% VARCUM 24-655/75% A-99 COMPOSITES
 IN HOT ACID

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rates in the two different regions were also observed to differ; 13 $\mu\text{g}/\text{cm}^2$ hr from 0 to 50 hours and about 1.5 $\mu\text{g}/\text{cm}^2$ hr from 150 to 2500 hours of aging in acid.

2.2 PHYSICAL PROPERTY MEASUREMENTS

2.2.1 Electrical Contact Resistance

The technique described previously* to measure electrical contact resistance was used for contacts between 32 wt% Varcum 24-655/68 wt% A-99 conductors and an FEP backing paper insert at 175°C. Figure II.5 shows the results. For the room temperature and the 175°C data, the effects of temperatures between 0.69×10^6 Pa (100 psia) and 1.38×10^6 Pa (200 psi) on contact resistance were slight. At pressures below 0.69×10^6 Pa the variations in the data are attributed to the low pressures and the limited number of data points. A definite hysteresis was observed when the pressure was reduced. The pressure for a stack at the bipolar plate rib - backing paper interface is estimated to be 0.965×10^6 Pa (140 psi), assuming uniform distribution of pressure across the ribs (Type C2C2, 1200 cm^2 bipolar plates).

At 175°C the equations for curves shown in Figure II.6 are:

$$r_3 = 1.75 \, 1/P + 0.480 \times 10^6 \quad (3)$$

(Correlation Coefficient = 0.96)

for increasing pressure, and

$$r_3 = 0.56 \, 1/P + 0.729 \times 10^6 \quad (4)$$

(Correlation Coefficient = 0.993)

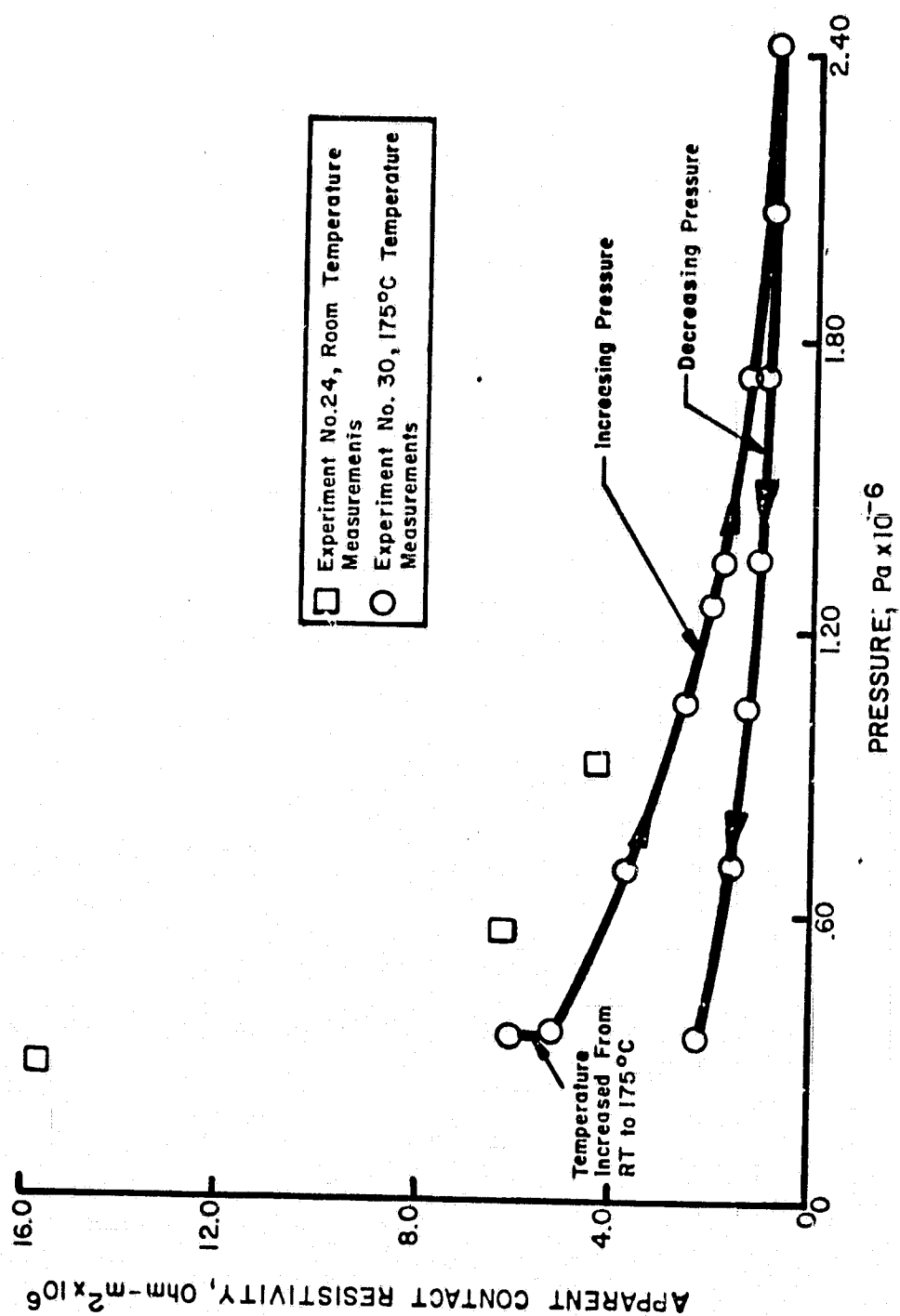
for decreasing pressure

Where,

P = pressure

r_3 = apparent electrical contact resistivity which is for two contacts and includes backing paper resistance.

* DEN3-67, No. 6, Jan.-Mar.1980.



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FIGURE II.5 ELECTRICAL CONTACT RESISTIVITY

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Based on current estimated values of the electrical resistivity of the backing paper, the changes in backing paper thickness and resistivity due to pressure variations are insignificant.

Future measurements will be performed with emphasis on pressure above 0.69×10^6 Pa (100 psi), and will use current production bipolar plate material (33 wt% Colloid 8440/67 wt% graphite). The conductor surfaces shall be as molded, not sanded.

2.2.2 Stack Resistance

Table II.4 updates* the calculated values for stack resistance and includes values for a stack with heat-treated bipolar plates. The resistance of a heat-treated bipolar plate is very small. The contact resistance of the heat-treated plates is also assumed to decrease and testing is planned to determine its magnitude.

* DEN3-67, No. 6, Jan. - Mar. 1980, pp 44-47.

TABLE II.4

INTERNAL RESISTANCE OF 5 - CELL, 1200 cm² STACKS

| LOCATION IN STACK | ESTIMATED RESISTANCE, mΩ | | COMMENTS |
|--|--------------------------|---------------------|---|
| | Current Plates | Heat-Treated Plates | |
| Plate | 0.78 | .01 | Values based on dimensions of C2C2 bipolar plates, and an electrical resistivity of 200 mΩ-cm for current plates. |
| Contact plate-backing interface | 0.47 | .47 | Values based on 37.4% FEP in backing paper, and 414 kPa (60 psia) on end plates. |
| In-plane backing paper | 0.08 | .08 | Values based on 37.4% FEP in backing paper #10,006. In-house resistivity measurement was 19 mΩ-cm. |
| Mat 1 Matrix | 0.44 | .47 | Parameter included: 180°C H ₃ PO ₄ and acid resistivity of 1.7 Ω-cm. |
| TOTAL | 1.77 | 1.03 | |
| Actual measurement on stacks, excluding current collectors | (Stack 428) 2.02 | (Stack 431) 0.95 | |
| Stack resistance accounted for: | 88% | 107% | |

*Electrical resistivity value was extrapolated from data in MacDonald, D. and Boyack, J., "Density, Electrical Conductivity and Vapor Pressure of Concentrated Phosphoric Acid," Journ. of Chem. and Eng. Data, 14, No. 3, July 1969.

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TASK III. ENDURANCE TESTING3.1 MEASUREMENT OF P_4O_{10} VAPOR CONCENTRATION

Previous acid loss experiments were performed under somewhat extreme cell conditions (a temperature of 195°C and an acid concentration of 104 to 110%). During this quarter, experiments were performed under conditions similar to actual cell operating conditions. The cells were kept at 180°C and humidified at a nominal temperature, 50°C . The partial water vapor pressures were calculated based on the amount of water collected and then acid concentrations were estimated at the humidity conditions. Of the three experiments performed, one showed a basic condensate rather than acidic condition, probably due to fouled air. For the other two, 0.24 ppm of P_4O_{10} at 300 cc/min of air flow, and 0.29 ppm at 110 cc/min of hydrogen flow were obtained at a cell temperature of 180°C . The estimated cell acid concentrations were 101 and 99%, respectively. Previously* the maximum P_4O_{10} level was reported to be 0.41 ppm at a cell temperature of 195°C with 107% acid.

3.2 EFFECT OF OPERATING VARIABLES ON CELL PERFORMANCE AND COMPONENT DEVELOPMENT

The most prominent development in 3-cell, 350 cm^2 stacks is the enhancement of stack performance using heat-treated plates. As expected, an almost 60 mV/cell gain was achieved by Stack 619 over Stack 603. Table III.1 illustrates performance characteristics of Stack 619 which was assembled with the newly developed, heat-treated plates and Mat-1 matrices. A stack performance of 673 mV/cell was achieved at 100 mA/cm^2 and 178°C . On an IR-free basis, the stack performed at 691 mV/cell.

* DEN3-67, No. 6, Jan.- Mar. 1980.

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TABLE III.1

ANALYSIS OF STACKS 619 and 603

| DESCRIPTION | | STACK 619 | STACK 603 |
|---|--|--------------|--------------|
| Performance, mV/cell | INITIAL, @ 100 mA/cm ² | 673 | 616 |
| | OCV | 850 | 790 |
| | AFTER 150 HRS: @ 100 mA/cm ² | 673 | 606 |
| | @ 200 mA/cm ² | 610 | 510 |
| | @ 400 mA/cm ² | 540 | -- |
| Resistance, mΩ | FOR TOTAL STACK | 3.0 | 4.3 |
| | FOR CELLS INCLUDING PLATES | 1.7 | 2.9 |
| | DUE TO COLLECTORS | 1.3 | 1.4 |
| Drop in Performance Due to Cell Resistance (IR), mV/cell | | 18 | 35 |
| Performance, IR-Free, mV/cell | | 691 | 655 |
| O ₂ Gain, mV | | 80 | 85 |
| Tafel Slopes, mV/decade | | 90 | 90 |

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At 400 mA/cm² an IR-free performance of 645 mV/cell was obtained. Stack 603 is also included in the table for comparison; it is one of the best long-term endurance stacks with similar components but with non-heat-treated plates. The initially superior performance of Stack 619 (by almost 60 mV) has continued throughout testing. The OCV reading has shown a comparable improvement over that of Stack 603.

An analysis of stack resistance is also included in the table. Stack 619 showed only a 17 mV/cell decrease in performance due to cell resistance, compared to Stack 603. The other major portion of gain appears to be due to increased platinum utilization of the electrodes. The fact that cells with non-heat-treated plates decrease in performance within a short time, and that O₂ gain and Tafel slopes do not differ significantly suggests that these plates might be leaching out species which have some effect on platinum utilization (poisons). The exact nature and mechanism of the performance loss with non-heat-treated bipolar plates is being investigated.

A similar performance gain was achieved in a full scale (5 cell, 1200 cm²) stack using the heat-treated plates (Stack 431, Table IV.1).

3.3 LONG-TERM COMPONENT EVALUATION

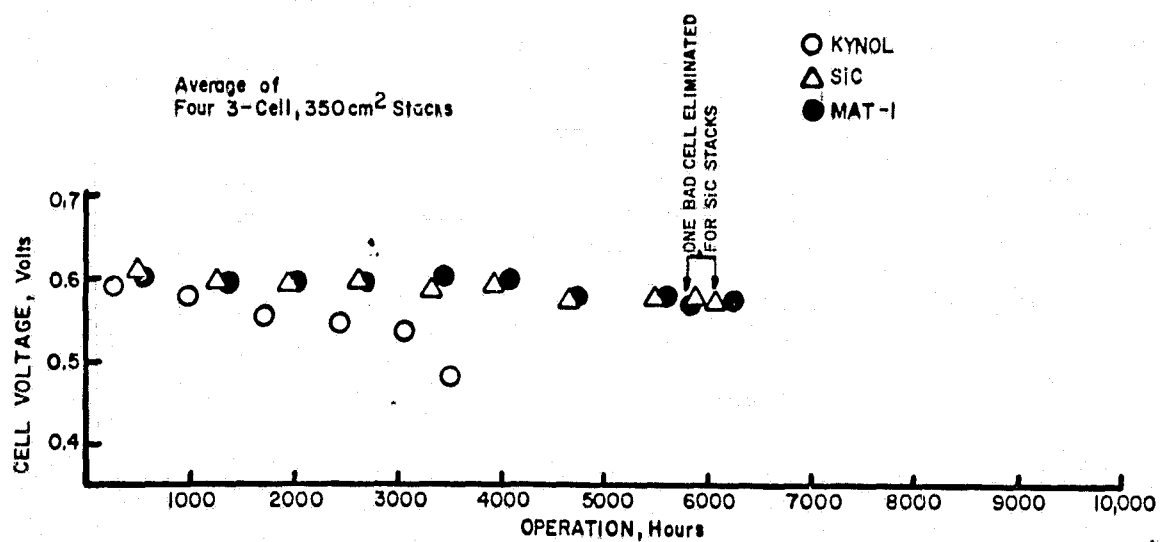
Long-term component evaluation of the twelve 3-cell, 350 cm² stacks started in September 1979 has continued. Of the twelve stacks, only eight are presently operating. All of the stacks with Kynol matrices (4 stacks) are shut down and have been disassembled. Performance characteristics of these stacks were discussed previously*. Disassembly analysis of these stacks indicated generally disintegrated phenolic fiber matrices. Samples have been taken for TEM, ECA and for other component measurements for further analysis.

* DEN3-67, No. 6, Jan.- Mar. 1980.

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Stacks with Mat-1 and SiC matrices continue to operate and indicate better stability and higher potential than the stacks with Kynol matrices. An update of the average cell performance of these endurance stacks with different matrices is presented in Figure III.1. The average life of SiC and Mat-1 stacks is now almost 6400 hours. Some of the stacks have developed soft plates at the acid channel area, causing gas sensitivity and performance loss. Note that one of the cells in the SiC matrix group is not included in the figure due to very poor performance.

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FIGURE III.1 ENDURANCE OF PAFC STACKS WITH DIFFERENT MATRICES

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TASK IV SHORT STACK TESTING

4.1 COMPONENT AND DESIGN TESTING

As reported in Section 3.2, the significant achievement in 5 cell, 1200 cm² size stack testing during the quarter is the scale-up capability of improved stack performance (Stack 619) using heat-treated plates. Stack 431 (Table IV.1) was assembled with heat-treated plates and Mat-1 matrices. An average performance of 665 mV/cell at 100 mA/cm² and 178°C was achieved in Stack 431. Similar performance was obtained for Stack 619, a 3-cell, 350 cm² stack (Section 3.2). Full scale stack performance is slightly lower than that of the medium size stack, possibly because of more temperature variations in the full-size stack.

A summary of short stack testing (5-cell, 1200 cm²) testing during the quarter is presented in Table IV.1. Stacks 415, 416, 417 and 422 were voluntarily terminated for disassembly. Stack 423 is sealed and stored at room temperature for further testing at a later date. Lifegraphs of Stacks 415 and 416 were presented previously*. Stack 415, assembled with sheet mold electrodes, performed at the same level (0.56V/cell) for more than 2800 hours and was terminated after 2997 hours of operation. This establishes the long-term operating capability of full-size sheet mold electrodes. A 3-cell, 350 cm² stack operated for more than 8600 hours with similar electrodes (Stack 379, not included in this report).

4.2 LONG-TERM COMPONENT EVALUATION IN FULL-SCALE STACKS

Three stacks (5-cell, 1200 cm² size) were assembled in May 1980 to establish the long-term operating capability of ERC's state-of-the-art components. These stacks, Nos. 428, 429 and 430, will be tested for up to 5000 hours. Stack

* DEN3-67 No.6, Jan.- Mar. 1980, pg. 54.

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TABLE IV.1 SUMMARY OF SHORT STACK TESTING

| STACK NO. | COMPONENT DESCRIPTION | | LIFE, Hours | PRESENT PERFORMANCE, V | | REMARKS |
|-----------|-----------------------|--------|-------------|-------------------------|------|---------------------------|
| | ELECTRODES | MATRIX | | @100 mA/cm ² | @OCV | |
| 415 | Sheet Mold | Kynol | 2997 | - | - | Disassembled |
| 416 | Rolled | Kynol | 2364 | - | - | Disassembled |
| 417 | Rolled | Kynol | 1753 | - | - | Disassembled |
| 422 | Rolled | Mat-1 | 395 | - | - | Disassembled |
| 423 | Rolled | Mat-1 | 1386 | - | - | Sealed for future testing |
| 424 | Sheet Mold | Mat-1 | 1896 | 0.56 | 0.81 | |
| 427 | Rolled | Mat-1 | 1266 | 0.55 | 0.79 | |
| 428 | Rolled | Mat-1 | 466 | 0.57 | 0.81 | |
| 429 | Rolled | Mat-1 | 422 | 0.57 | 0.81 | |
| 430 | Rolled | Mat-1 | 333 | 0.58 | 0.82 | |
| 431* | Rolled | Mat-1 | 138 | 0.67 | 0.88 | |

* Stack contains heat-treated plates.

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components are as approved by the NASA/Lewis program manager. Mat-1 matrices are used in all the stacks and the 'wet assembly' procedure was followed. Gold-plated copper current collectors with central posts are used. Performance of these stacks is noted in Table IV.1. Note that an almost 90 to 100 mV/cell gain is achieved by using heat-treated plates in Stack 431.